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ON THE ACCURACY OF THE ROSIWAL METHOD FOR THE DETERMINATION OF THE MINERALS IN A ROCK

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Some years ago Julien¹ expressed a doubt as to the accuracy of the Rosiwal method in the determination of the percentage composition of rocks in general. He thought, if the fragments were equicubical, that d , the measured average diameter of the mineral plates, might give correct results, but for unequicubical grains the value d^3 should be used, and for rounded grains,² thick lenses, or short spindles, $.7854\sqrt[3]{a}$ or d^2 . For schistose rocks with flakes and interlaminae parallel and of equal thickness, he suggested d^2 , for prisms or blades with parallel interlaminae, $d' = \sqrt[3]{l \times b \times t}$, where l , b , and t are the length, breadth, and thickness of plates.

Williams,³ several years later, studied the comparative accuracy of various methods, using a pink granite from Westerly, Rhode Island, with grains ranging from 1.5 to 5 mm. in diameter. Several determinations were made in each experimental method and the results were averaged and tabulated with the calculated results as shown in Table I.

The first column is calculated from the chemical analysis and is practically the norm, except for the small amount of biotite. The second column represents the percentages of the different minerals obtained by separation according to their specific gravities in Sonstadt's solution. The agreement of this determination with the preceding is fairly close. The third column is based upon

¹ A. A. Julien, "Genesis of the Amphibole Schists and Serpentes of Manhattan Island, New York," *Bull. Geol. Soc. Amer.*, XIV (1902), 460-68.

² Julien gives $.7854\sqrt[3]{a}$ or d^2 for this value. Williams gives $.5236d^3$, the volume of a sphere, as Julien's (possibly later) figure.

³ Ira A. Williams, "The Comparative Accuracy of the Methods for Determining the Percentages of the Several Components of an Igneous Rock," *Amer. Geol.*, XXV (1905), 34-46.

microscopic measurements of the diameters of grains in thin section made according to the Rosiwal method, the results thus obtained being multiplied by the respective specific gravities to give weight percentages. (Personally the present writers prefer to transfer the specific gravity determinations into volume percentages rather than the reverse, since one estimates the composition of a rock by volumes and not by specific gravities.) The fourth column represents six different photomicrographic prints, made of different parts of the slide, which were dissected and weighed according to the Delesse-Sollas-Joly method. The weights of the fragments of paper were assumed to be proportional to the areas, therefore to the

TABLE I

	I Calculated from Chemical Analysis	II Heavy Solution	III Measure- ments of Diameters Rosiwal Method	IV Photomi- crographs of Areas	V Areas (d^2) Measured by Microm. Ocular	VI Calcula- tions of d^3 from Column III	VII Calcula- tions of d^3 from Column V
Quartz	35.90	34.25	19.02	23.23	19.90	1.41	12.14
Orthoclase	34.48	32.47	43.65	43.64	43.67	56.04	49.92
Plagioclase	24.99	26.91	32.90	32.90	32.90	42.24	37.31
Mica	0.26						
Magnetite	2.60	5.68	4.43	0.25	3.52	0.31	0.63
Hematite	1.10						

volumes of the minerals. The percentages were computed as before. The fifth column represents measurements of areas made by means of a net-micrometer ocular, as originally used by Rosiwal; the number of squares included within the boundaries of the different minerals being counted. The areas again were assumed to be proportional to volumes. The sixth column was obtained by cubing the values in column III and reducing the sum to 100. The last column represents d^3 calculated from the value of d^2 in column V.

Williams pointed out the close agreement between the composition calculated from the chemical analysis and the results obtained with Sonstadt's solution on the one hand, and that obtained by the Rosiwal method and the measurement of areas on the other. The two sets of values, however, do not agree. He remarked that "though the absolute volumes (of the microscopic methods) may not be correct, using d as indicated, relative volumes as expressed

in percentages may nevertheless give as significant results as if the actual value of d were known." The results of columns VI and VII indicate that d^3 cannot be used for calculating the percentage composition when d is the measured width of the mineral grains in the thin section.

Williams concludes that

any one of the direct measurements with the microscope does not appear to fulfill even approximately the necessary conditions for a statement of the complete quantitative composition," but that "heavy solutions are a convenient and readily applicable method of separating the minerals of a rock and may be valuable in determining quantitative relations. . . . The accuracy of the results will be directly proportional to the coarseness of grain and the readiness with which the minerals can be separated.

So far as the writers are aware, no other comparisons have been made between the specific gravity method and that of Rosiwal. Contrary opinions to those just quoted are held by many petrographers. Thus in Table II we have the comparison of a

TABLE II

	I Calculated Composition from Analysis	II Rosiwal Volume Analysis: First Specimen	III Weight Percentage: First Specimen	IV Rosiwal Volume Anal.: Second Specimen	V Weight Percentage: Second Specimen
Quartz	22.86	23.17	22.55	20.97	20.34
Orthoclase	18.35	18.62	17.57	21.07	19.82
Ab ₃ An ₂	39.74	43.10	42.47	42.20	41.48
Biotite	10.92	8.87	9.77	8.15	8.94
Hornblende }	3.56	3.78	4.44	4.84	5.67
Pyroxene }		1.97	2.37	2.07	2.50
Magnetite	1.86	.40	.76	.54	1.02
Pyrite, etc.	1.86	.04	.07	.02	.04

computation from chemical data of mineral composition with measured linear dimensions according to the Rosiwal method made by Cross, Iddings, Pirsson, and Washington¹ for the Butte "granite."

In Table II, columns I, III, and V should be compared. The authors state that a small amount of pyroxene was intergrown with

¹ Cross, Iddings, Pirsson, and Washington, "A Quantitative Chemico-Mineralogical Classification and Nomenclature of Igneous Rocks," *Jour. Geol.*, X (1902), 681-82.

Ibid., *Quantitative Classification of Igneous Rocks* (Chicago, 1903), p. 286.

the hornblende and was probably included in the analyzed hornblendic material. The two minerals are, therefore, calculated together. There can be no question about the success of the Rosiwal determinations in this case. Cross, Iddings, Pirsson, and Washington¹ say: "It is possible . . . to determine very closely the proportions of the minerals present. . . ." This determination proves either the accuracy of the Rosiwal method or, since it is only a single example, an almost exact compensating error.

Among other petrographers who have found the microscopic methods of sufficient accuracy are Barrell,² who determined his constituents by a somewhat simpler, though less accurate, method. He says:

The field of view of the microscope is divided into quadrants by the cross-hairs, and these are mentally divided into sectors which are thirds, fifths, or smaller fractions. By taking such a power of objective that the component to be estimated is represented in the field of view by a fair number of crystals, the latter can be mentally collected together and packed into one quadrant, and the fraction which it fills estimated. . . . In the case of holocrystalline rocks, where the writer has been enabled to check this method of estimating the mineral and chemical composition by comparison with laboratory analyses, *it is found that the error in any one element should be less than 1 per cent and is often less than one-half per cent.* [The italics are ours.]

Robinson³ compared the Rosiwal measurements of a porphyritic dacite with the composition calculated from an analysis and found that

there is a difference of 3.2 per cent between the total salic components, including the excess of alumina in the calculated mode, and of 2.6 per cent between the dark components. . . . The comparison will give an idea of the accuracy of the Rosiwal method as applied to porphyritic lavas containing small amounts of minerals of highly variable size.

Tyrrell⁴ says:

The graphic method of quantitative mineral measurement invented by Rosiwal yields sufficiently accurate results for classificatory purposes in the

¹ *Ibid.*, p. 204.

² Joseph Barrell, "Microscopical Petrography of the Elkhorn Mining District, Jefferson County, Montana," XXII An. *U.S. Geol. Surv.*, Pt. II, 1901, pp. 512-13.

³ Henry Hollister Robinson, "The San Franciscan Volcanic Field, Arizona," *U.S. Geol. Surv., Prof. Paper* 76, 1913, p. 121.

⁴ G. W. Tyrrell, "A Review of Igneous Rock Classifications," *Science Progress*, 1914, pp. 74-75.

great majority of types. . . . Notwithstanding the opinion of Williams, the Rosiwal method of estimation is capable of giving extremely accurate results. The writer . . . has found the chemical composition calculated from the results of the Rosiwal measurements to be strikingly accordant with that obtained by ordinary chemical analysis.

In view of Williams' apparently careful study and rejection of the Rosiwal and other microscopic methods, and of the increasing use made of volumetric determinations of rock constituents by other petrographers, it was thought desirable to repeat the experiments with all possible precautions against errors in both the Rosiwal and the heavy solution methods. Williams' study is apparently the only one in which there was a comparison of the percentages determined by the microscopic measurement method and an actual separation of the minerals; all others are comparisons between the former and recalculated chemical analyses, with all the possible errors in allotting constituents due to the uncertain composition of the dark constituents and the possible soda content of the orthoclase.

The material chosen for the experiment by the present writers was the Butte "granite." It was selected because it had been chemically analyzed, because there was available ample material from the original locality, and because there were on hand six thin sections and the two determinations by the Rosiwal method made by others as mentioned above, and five determinations by students on one slide (Table III, column I). Furthermore, the material is of fairly uniform grain and composition in widely separated localities, thus giving a fairer test than would a rock differing possibly in different sections and thus probably different from the material analyzed.

The six different sections were measured by the Rosiwal method from one to five times each, fourteen determinations being made in all. The average of each slide, reduced to weight percentages, is shown below. In Table III the first six columns represent the six sections; the number in parentheses after each indicates the number of times it was measured. The seventh and eighth columns are the determinations taken from Cross, Iddings, Pirssons, and Washington and quoted above. The variations between the read-

ings obtained for the different sections are possibly due to differences in the sections themselves, the grain of the rock being coarse enough to show slight variations. The last column shows the average of the sixteen determinations, and not the average of the averages.

TABLE III

	I (5)	II (1)	III (2)	IV (3)	V (1)	VI (2)	Cross, Iddings, Pirsson, and Washington		Average
Quartz.....	20.31	22.4	23.79	24.76	20.3	17.64	22.55	20.34	21.6
Orthoclase.....	25.31	31.6	18.15	30.77	28.5	29.11	17.57	19.82	25.8
Plagioclase.....	38.75	32.9	43.00	25.18	32.3	32.01	42.47	41.48	35.6
Biotite.....	8.98	8.3	8.35	9.70	10.8	10.47	9.77	8.94	9.2
Hornblende.....	4.65	4.4	5.81	7.02	5.2	7.47	4.44	5.67	5.6
Augite.....	.70	.2	1.01	1.3	2.76	2.37	2.50	1.1
Magnetite.....	.7463	1.50	1.2	.58	.76	1.02	.8
Apatite.....	.03	.1	.100
Titanite.....	.04090
Pyrite.....	.5504	0.3	.09	.07	.04	.2
Zircon.....	.03
	99.99	99.9	99.96	99.94	99.9	100.13	99.98	99.81	99.9

For the separation of the constituents by mechanical means, several methods were employed. The magnetite was first removed by means of an electromagnet.¹ By repeating the process the material removed was practically pure. The biotite was next separated by Rosenbusch's² method of shaking the rock powder from an inclined sheet of semi-glazed paper. A remarkably clean separation resulted as shown by microscopic examination, only a few flat hornblende flakes being included as impurities. The remaining material was now placed in a Brögger-Harada³ tube with about a half-pint of Sonstadt's (potassium-mercuric-iodide) solution, and enough water was added to bring down the hornblende and augite. For the purpose of these experiments it was not necessary to separate these two minerals, so it was not attempted. With these minerals the zircon, apatite, and titanite came down. Thus far the separation was easy; the difference in color between the heavy minerals and those remaining clearly showed when

¹ The instrument is illustrated and the method described in Johannsen's *Manual of Petrographic Methods*, 1918, Fig. 725, and p. 540.

² *Ibid.*, p. 557.

³ *Ibid.*, p. 551.

the separation was complete. The separation of the plagioclase (Sp. gr. 2.68) from the quartz (Sp. gr. 2.65) was the most difficult. As an indicator, therefore, a small Herkimer County quartz crystal was placed in the solution, which was then diluted until the crystal began to sink, after which a very small amount of concentrated solution was added to make it slowly rise to the top. This adjustment between the specific gravity of the quartz and that of the liquid is here so delicate that a slight change in the density of the solution, such as results from a slight change in the temperature of the room, caused the quartz, which has a very small coefficient of expansion, to settle during the night and rise again during the day. When the equilibrium was established, the plagioclase was removed, washed, dried, and weighed as in the other cases. The quartz was brought down by adding enough water to bring down the indicator. The final material was the orthoclase.

The greatest objection to the specific-gravity method seems to be the impossibility of preparing powder which consists of homogeneous material. In many cases material of one kind will cling to grains of another and will increase or decrease their specific gravities. The small amount of mixed material shows up very clearly in the separation, especially when between light and dark minerals, for the mixed heavier grayish material will come down before the pure white mineral, or, on the other hand, the precipitated dark mineral will be covered with a thin layer of material that is grayish. The probabilities are that the two errors compensate. The material should be crushed fine enough to separate each constituent from every other, but not so fine as to produce dust, since some minerals pulverize much more readily than others and a different proportion of each will be lost, destroying the accuracy of the result. A happy medium must therefore be taken between purity and selective sieving, since absolute purity cannot be expected. Microscopic examination of a small amount of the material of a certain size will readily make it possible to determine whether or no the rock has been crushed fine enough to produce a good separation of the constituents. For the following experiments various-sized grains were used, as indicated at the top of each column in Table IV. After crushing and sifting, the total samples weighed

140-150 gms. This was quartered, and one-fourth of each quarter was used to make two samples for determination. For this particular rock, grains passing through a 60-mesh sieve and remaining on a 100 mesh seemed to be very suitable.

TABLE IV

	I Sp. gr.	II Mesh 80-100	III Mesh 80-100	IV Mesh 60-100	V Mesh 60-100	VI Mesh 60-100	VII Mesh 60-100	VIII Average
Magnetite.....	5.10	1.38	1.09	1.42	1.12	1.36	1.30	1.26
Biotite.....	3.00	11.10	9.49	9.94	9.74	9.88	9.72	9.98
Hornblende.....	3.20	6.66	6.93	6.66	6.44	6.31	6.25	6.54
Augite.....	3.30							
Plagioclase.....	2.68	37.87	37.10	34.60	31.65	32.82	33.40	34.57
Quartz.....	2.65	19.21	21.11	21.35	23.22	24.65	23.70	22.21
Orthoclase.....	2.57	23.07	24.25	26.00	27.06	25.02	26.06	25.25
		99.29	99.97	99.97	99.23	100.04	100.43	99.81

Comparing now the final results of each of the three methods, we have the results given in Table V.

TABLE V

	Calculated from Analysis	Weight Percentage Roswal	Heavy Solution
Quartz.....	22.86	21.6	22.21
Orthoclase.....	18.35	25.8	25.24
Plagioclase.....	39.74	35.6	34.57
Biotite.....	10.92	9.2	9.98
Pyribole.....	3.56	6.7	6.54
Magnetite.....	1.86	.8	1.26
Pyrite, etc.....	1.86	.2
	99.15	99.9	99.83

The amount of quartz in the calculated analysis is practically the same as in the specific-gravity determination, and the Roswal value is only slightly less.

The orthoclase is low in the calculated analysis and practically the same in columns 2 and 3, while the calculated plagioclase is high as compared with the other two determinations. This is unquestionably due to the fact that the orthoclase carries soda. In the computation of the analysis by Cross, Iddings, Pirsson, and

Washington (p. 225), orthoclase was taken as $K_2O \cdot Al_2O_3 \cdot 6SiO_2$ in the place of the actual composition. In all probability the orthoclase-plagioclase percentages of the Rosiwal and specific-gravity determinations are more nearly those of the actual minerals present than those computed from the chemical analysis. It will be seen that the sums of the feldspars, 58.09, 61.4, and 59.81, are not greatly different.

The sums of the leucocratic constituents in the three columns, 80.95, 83.00, and 82.02, are also close together, as are the biotite and magnetite values.

The only noteworthy variations in any of the determinations are in the values of the remaining constituents—the pyribole and the pyrite. Here the values for the former are 5.42, 7.5, and 6.54, and for the latter, 1.86, 0.3, and 0. For the sum of the two constituents in each column, however, we have 7.28, 7.8, and 6.54. In the first place the sum of all the constituents in the analysis calculated by Cross, Iddings, Pirsson, and Washington in column 1 is only 99.15, as against 99.9 and 99.81 in the other two columns; consequently the first-column values should be slightly increased. The calculated amount of pyrite (1.86) is greatly in excess of that seen in any slide or in the hand specimen. The fraction of 1 per cent given by the thin section is much more nearly correct. When the greater part of the pyrite is added to the pyribole in column 1, the values are more nearly the same. In the specific-gravity determination the magnetite and biotite were removed before the pulverized material was placed in the Sonstadt solution, which was then diluted until the pyribole and the small amounts of pyrite, apatite, and zircon present came down. Therefore these latter minerals are included with the pyribole in that column. The rock analysis and the thin sections were clearly from material slightly more melanocratic than the pulverized rock used in the specific-gravity determination, as is shown by the difference between the leucocratic-melanocratic ratio of columns 1 and 2 as against column 3.